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Dynamics of charge states at the surface of a ferroelectric nanoparticle in a liquid crystal

J.M. Gudenko[1], O.S. Pylypchuk[1], V.V. Vainberg[1,*], V.N. Poroshin[1], D.O. Stetsenko[1], I.A. Gvozdovskyy[1], O.V. Bereznykov[1], S.E. Ivanchenko[2], E.A. Eliseev[2,*], A.N. Morozovska[1,*]**

[1]Institute of Physics, National Academy of Sciences of Ukraine,46, Nauky Ave., 03028 Kyiv, Ukraine
[2]Frantsevich Institute of Materials Science, National Academy of Sciences of Ukraine,
3, Omeliana Pritsaka str., 03142 Kyiv, Ukraine
Corresponding author e-mail: viktor.vainberg@gmail.com*, eugene.a.eliseev@gmail.com**, anna.n.morozovska@gmail.com***

Abstract. The liquid crystal with suspended ferroelectric nanoparticles is an interesting object for fundamental research on long-range dipole-dipole interactions. Additionally, it is promising for optical, optoelectronic and electrochemical applications. These suspensions can serve as basic elements for advanced nonvolatile memory cells and energy storage devices. The work studies cells filled with a nematic liquid crystal 5CB and cells containing 5CB with 0.5 and 1 wt.% of $BaTiO_3$ nanoparticles with an average size of 24 nm. We analyzed the time dependences of the current flowing through the cells at a constant applied voltage and the voltage dynamics in the no-load mode. The time dependences of the current and voltage show a slowing-down decay rate. For the cells with $BaTiO_3$ nanoparticles, the decrease in the time decay is characteristic. A possible physical reason for the retarding time decay is the indirect effect of screening charges, which cover ferroelectric nanoparticles, and slow ionic transport in the liquid crystal. To explain the dynamics of current and voltage, the finite element modeling of the polarization distribution, domain structure dynamics, and charge state of nanoparticles in a liquid crystal is performed using Landau–Ginzburg–Devonshire approach. Theoretical results confirmed the leading role of screening charges, because the surface of a ferroelectric nanoparticle adsorbs an ionic-electronic charge that partially screens its spontaneous polarization in single-domain and/or poly-domain states. When an electric field is applied to the liquid crystal with nanoparticles, it can release part of the screening charge (mainly due to the change in the polarization of the nanoparticle), which will lead to a decrease in the decay rate of the current and voltage dependences.

Keywords: ferroelectric nanoparticles, charge states, ionic-electronic screening, ferro-ionic coupling, liquid crystal.

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1. Introduction

Studies of the unique electrophysical properties of ferroelectric nanoparticles with sizes varying from 5 to 50 nm are of considerable fundamental and practical interest [1–5]. It is worth highlighting several studies of ferroelectric $BaTiO_3$ nanoparticles covered by heptane and oleic acid, whose structure becomes a core-shell type structure [6–10]. In these studies, nanoparticles suspended in a liquid dielectric medium, unexpectedly have a "giant" spontaneous polarization, which reaches 1.3 C/m^2 at room temperature. This value exceeds the spontaneous polarization of a $BaTiO_3$ single crystal by more than 5 times. Core-shell nanoparticles with "giant" spontaneous polarization can significantly enhance the electro-caloric response [11] of colloids and influence the phase transitions dynamics in nematic liquid crystals [6–10].

A significant enhancement of spontaneous polarization in small $BaTiO_3$ nanoparticles can be caused by both the strain gradient and/or the curvature of their surface (since the ferroelectric is very sensitive to various types of deformations), and by the electrochemical interaction of the particle core with the shell and/or the surrounding environment. Particularly, it has been demonstrated experimentally [12] that the spontaneous polarization P_S of $BaTiO_3$ films increases by 250% under epitaxial strain (lattice mismatch), while the Curie temperature T_C increases to approximately 500 °C. First-

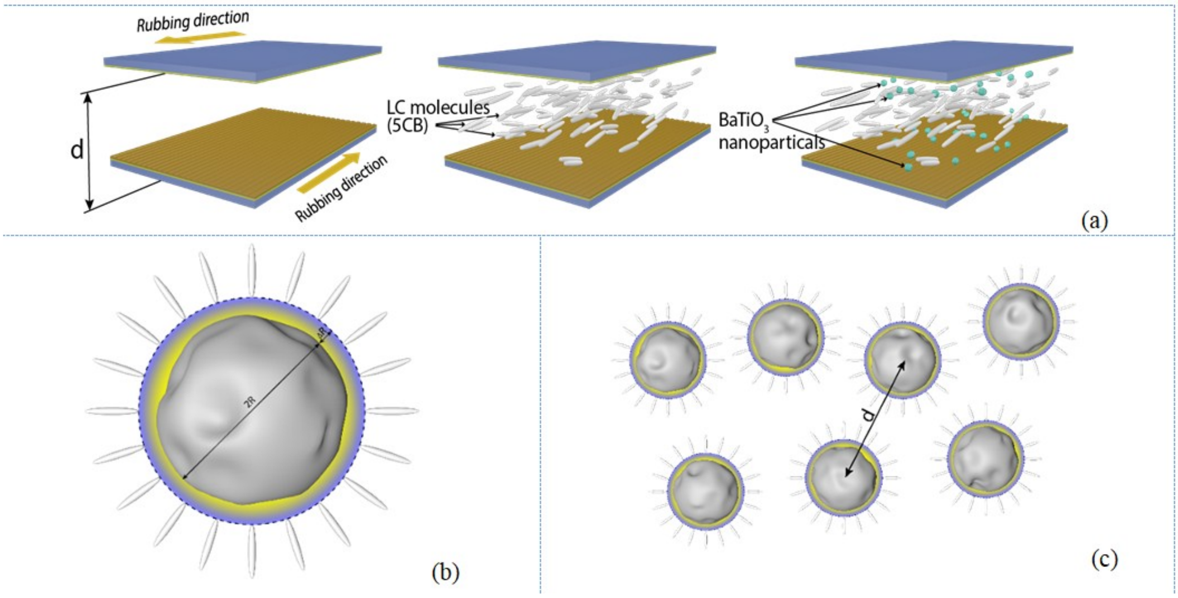

Fig. 1. (a) Schematic view of the studied samples with pure LCs and with LCs suspension. (b) A $BaTiO_3$ core with an average size 2R covered by a screening shell of ionic-electronic charge. (c) The core-shell nanoparticles in a liquid crystal. Note that $2R \sim 25$ nm, and the length of the liquid crystal molecules is close to 2–5 nm.

principles calculations [13] and thermodynamic theory [14] explain the experimental results. It was subsequently shown that electrochemical strains are responsible for the strong increase in the Curie temperature (above 167 °C) and tetragonality (up to 1.032) near the surface of $BaTiO_3$ films with oxygen vacancies [15]. The lattice constant mismatch, which causes epitaxial and/or core-shell strains, is responsible for the enhancement of P_S, T_C, and tetragonality in the core-shell $BaTiO_3$ nanoparticles [16–18].

The issue of the electrochemical interaction of the nanoparticle core with its shell and/or the surrounding environment remains largely unexplored. In this context, investigation of the electrophysical properties of small $BaTiO_3$ nanoparticles suspended in a liquid crystal is of significant scientific interest. Liquid crystals with suspended $BaTiO_3$ nanoparticles are promising for optical, optoelectronic and electrochemical applications. These suspensions can serve as basic elements for advanced nonvolatile memory cells and energy storage devices [19].

2. Analysis of experimental results

We prepared cells filled with pure nematic liquid crystal 5CB and liquid crystal suspension containing 0.5 and 1 wt.% of $BaTiO_3$ nanoparticles with an average size of 24 nm. A schematic illustration of the cells preparation is presented in Fig. 1a. A schematic illustration of a $BaTiO_3$ nanoparticle, covered with a shell of screening ionic-electronic charge and surrounded by elongated polar molecules of the liquid crystal, is presented in Fig. 1b.

The study of electric transport properties in the liquid crystal 5CB with suspended $BaTiO_3$ nanoparticles is focused on the time transient processes of electric transport caused by polarization effects and charge accumulation in the samples under different conditions. For this purpose, we carried out measurements of the current *versus* time in the cells after applying a constant electric voltage of various magnitudes across the sample. Additionally, the time decay of the voltage across the sample in the no-load regime after switching off the power

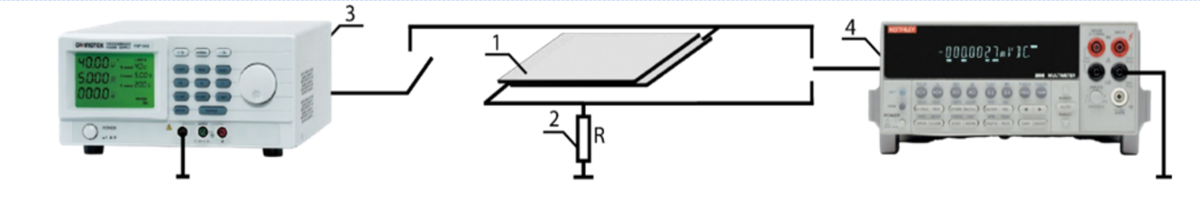

Fig. 2. Principal scheme for electrical measurements: *1* – a liquid crystal cell with or without nanoparticles, *2* – a series resistor R for measuring current through the sample, *3* – power supply “GW Instek PSP-603”, *4* – voltmeter “Keithley 2000 digital multimeter”.

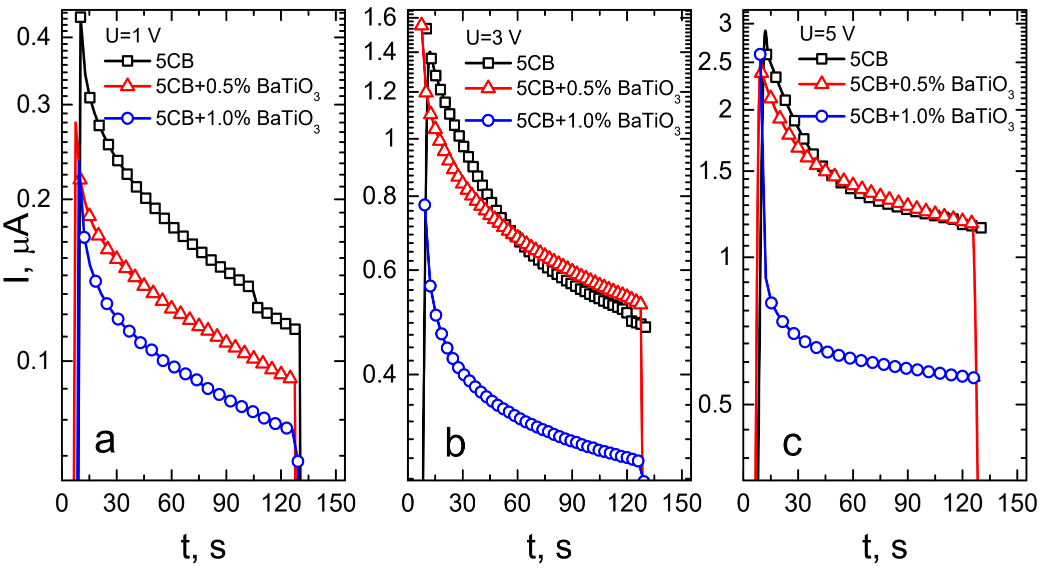

Fig. 3. Time dependences of the current decay through the cells after applying the constant voltage across them. Applied voltages are 1 V (a), 3 V (b) and 5 V (c).

supply was measured. For comparison, pure 5CB liquid crystal cells without nanoparticles were also studied.

The main part of the electrical scheme for measuring the transient characteristics is shown in Fig. 2.

The measuring technique is the following. A voltage of a constant magnitude is applied across the cell, and the decaying current through the cell is measured during 120 s. The current is determined from the voltage drop across the series resistor R. The voltage was recorded using a digital multimeter, Keithley 2000, connected to the computer. After that, the power supply is switched off, the switch to the no-load regime occurs, and the time decay of the voltage across the sample is recorded. To remove the residual charge from the sample after completing each measurement, the end contacts of the samples are short-circuited until the total voltage drops down to 0.

Fig. 3 shows the time dependences of the current decay through the samples after applying a constant voltage across them for the suspension of 5CB with 2 concentrations of dispersed BaTiO$_3$ nanoparticles, and pure 5CB. For all samples, one can see the long-lasting transient processes with a duration of several hundred seconds. The total duration of decay is approximately the same in all samples, and the curves do not obey a simple exponential law. The difference between the curves is mainly in the magnitude of the current and is determined by the concentration of nanoparticles.

The first set of the curves obtained at the voltage magnitude of 1 V is replotted in Fig. 4 in the coordinates $\ln\left(\ln\left(J_0/J\right)\right)$ and $\ln\left(t-t_0\right)$. Here, J_0 and t_0 are the current magnitude and time at the beginning of the current decay curve, respectively. It is seen that all curves are very well fitted by the linear behavior in these coordinates, which evidences that they obey the stretched-exponent law:

$$J = J_0 \exp\left[-\left(\frac{t-t_0}{\tau}\right)^{\beta}\right] + a. \qquad (1)$$

We neglect the value of a compared to the total range of variation of the current J. The stretched-exponent law is characteristic for transient processes in the spatially disordered systems, and, in our case, it may evidence both the non-uniform structure of the 5CB suspension and the non-uniform distribution of the ferroelectric nanoparticles. From the curves in Fig. 4, one can obtain β to be approximately 0.3 – 0.32 and τ to be 260 to 300 s.

Fig. 5 shows the time dependences of the voltage decay in the cells in the no-load regime after switching off the power supply. It is seen that decay does not obey

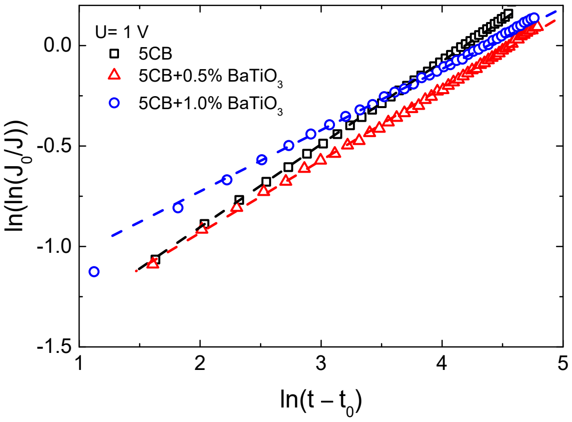

Fig. 4. Fitting of the current decay curves by the stretched-exponent law. The fitted curves (dashed lines) and measured points (symbols) are shown for the voltage drop of 1 V across the sample. The symbols designating samples correspond to those in Fig. 3a.

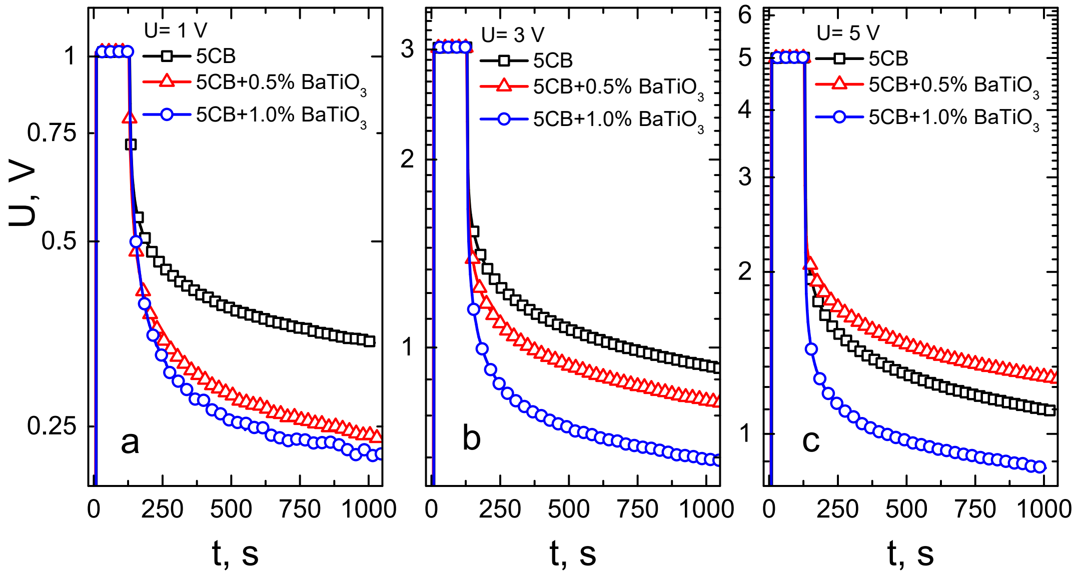

Fig. 5. Time dependences of the voltage decay in the samples in the no-load regime after switching off the power supply. The maximum applied voltages are 1 V (a), 3 V (b) and 5 V (c).

the simple exponential law. Instead, its behavior is more complicated compared to the current decay during the charging process. In general, immediately after switching off the power supply, one can observe a sharp decrease in the voltage, which may be related to the rapid polarization. Then the samples with dispersed ferroelectric nanoparticles and without them manifest somewhat different behavior. The pure 5CB cell comes into approximately single-exponential decay, the cells with suspended nanoparticles demonstrate the decay, which overlaps the quick decaying part and a considerable part of the "next" decay. This behavior may be described by the stretched-exponent law (Fig. 6), after which the decay curve deviates to a less steep decay with almost a single-exponential behavior.

3. Analysis of theoretical results

To clarify the physical reason for the time dependences of current and voltage, the numerical simulation of the electrophysical state of suspensions was carried out by the finite element modeling (FEM) using the phenomenological Landau–Ginsburg–Devonshire (LGD) approach for the calculations of particle polarization and its domain structure dynamics, electrostatic equations for the self-consistent calculations of electric field, and elasticity theory for the modeling of the elastic strains and stresses. Details of the calculations, the system of nonlinear LGD equations with boundary conditions, and material parameters of $BaTiO_3$ are given in Ref. [11].

The simulation assumes that the surface of a polarized ferroelectric nanoparticle accumulates an ionic-electronic charge, which screens partially or completely the electric polarization outside the particle. The density of this charge depends on the state of the polarization at the nanoparticle surface and on the properties of the surrounding liquid medium [20]. The ionic-electronic charge density determines the effective screening length λ of the spontaneous polarization at the nanoparticle surface.

The Stephenson–Highland model [21] describes the relationship between surface charge density $\sigma_S[\delta\phi]$ and electric potential excess $\delta\phi$ at the nanoparticle surface. This model accounts for the coverages of positive and negative surface charges (*e.g.*, ions, anions, and electrons) in a self-consistent manner. The corresponding Langmuir adsorption isotherm is given by expression [22]:

$$\sigma_S[\delta\varphi] \cong \sum_i \frac{eZ_i}{A_i}\left(1 + a_i^{-1}\exp\left[\frac{\Delta G_i + eZ_i\,\delta\varphi}{k_B T}\right]\right)^{-1}, \tag{2a}$$

where e is the electron charge, Z_i – ionization number of the adsorbed ions, a_i – dimensionless chemical activity of ions in the environment (as a rule $0 \le a_i \le 1$), T – absolute temperature, A_i – area per surface site for the adsorbed ion, and ΔG_i are the formation energies of the positive (subscript $i = 1$) and negative (subscript $i = 2$) surface charges (*e.g.*, ions and/or electrons) at normal conditions.

Linearization of Eq. (2a) with respect to the electrostatic potential gives the expression for the effective screening length λ associated with the adsorbed charges [20]:

$$\frac{1}{\lambda} \approx \sum_i \frac{(eZ_i)^2 a_i \exp\left[\frac{\Delta G_i}{k_B T}\right]}{\varepsilon_0 k_B T A_i\left(a_i + \exp\left[\frac{\Delta G_i}{k_B T}\right]\right)^2}. \tag{2b}$$

For relatively large λ (more than 10 nm), the polarization screening is weak, and the nanoparticle is in the non-polar paraelectric phase induced by its small size (~25 nm). With decreasing λ (from 10 to 1 nm), the screening increases, and the spontaneous polarization of the nanoparticle remains. However, the screening is not strong enough to maintain the single-domain polarization state of the nanoparticle. Since barium titanate is a multiaxial ferroelectric, the polarization of the nanoparticles can rotate, forming the vortex-like structures that significantly reduces the depolarization field inside the particle and the scattering field outside them [23]. A single-domain state of the polarization inside the particle is energetically preferable at very small λ (much less than 1 Å). "Perfect" screening ($\lambda \rightarrow 0$) minimizes the indirect electrostatic interaction between the nanoparticles *via* the liquid crystal and the direct interaction of the particles with the liquid crystal.

Thus, the analysis of the polarization state of a nanoparticle at large and small λ allows for an explanation of the experimental observations. Therefore, the FEM was carried out for λ within 0.01…10 nm, which provides a significant influence of the ionic-electronic charge on the ferroelectric polarization of the particle ("ferro-ionic" coupling) and does not exclude long-range electrical interactions between the particles with increasing λ. We also assume that λ significantly increases with decrease in the nanoparticle concentration, since the immersion of nanoparticles covered by surfactant leads to the appearance of additional ionic-electronic charge in the dielectric liquid crystal 5CB.

The voltage dependence of the charge density on the electrodes of a cell with a liquid crystal 5CB and densely packed 24-nm spherical $BaTiO_3$ nanoparticles (particle concentration is more than 50 vol.%) is shown in Fig. 7a. The electric polarization of the particles is screened by the ionic-electronic charge adsorbed at their surface. FEM was carried out at room temperature for the radius of the nanoparticles $R = 12$ nm, the screening length $\lambda = 0.01$ nm (Fig. 7a), 0.1 nm (Fig. 7b), 1 nm (Fig. 7c) and 10 nm (Fig. 7d).

The insets in Fig.7a show the distributions of spontaneous polarization P_S inside the $BaTiO_3$ nanoparticle and the electric potential φ inside and around the particle in the states "+1" and "–1", which are indicated on the charge loop and corresponds to zero voltage. The nanoparticles are divided into domains, and the direction of polarization inside the domains is determined by the direction of the voltage sweep ("pre-history"). The polarization of the domains in the states "+1" and "–1", corresponding to opposite directions of the voltage sweep, is opposite. The interaction of the polarization of domains with the liquid crystal and with other nanoparticles leads to a specific potential distribution, which is also inverted in the states "+1" and "–1". Since the single-domain state is not observed at low voltages, the hysteresis loop of the charge stored by the liquid crystal cell is narrow and inclined, and the maximal spontaneous polarization in the "+1" and "–1" states (~ 0.06 C/m^2) is approximately 4 times smaller than the polarization of a bulk barium titanate single-crystal at room temperature (~ 0.25 C/m^2).

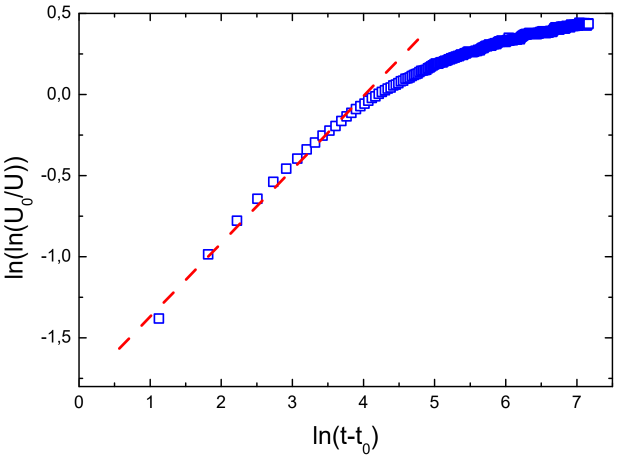

Fig. 6. Fitting of the voltage decay curve for the liquid crystal 5CB with 1 wt.% of $BaTiO_3$ nanoparticles by the stretched-exponential law.

Figs 7b–7d show the charge density dependences on the electrodes covering the cell with a suspension of liquid crystal with 10, 1, and 0.1 vol.% of $BaTiO_3$ nanoparticles, respectively. The charge density dependences are characterized by very weak hysteresis, mainly associated with the polarization dynamics of the liquid crystal organic molecules. An increase in the concentration of $BaTiO_3$ nanoparticles leads to the appearance of small additional "loops" or "self-intersections" located symmetrically at the ends of the main loop of the voltage-charge characteristic. The nanoparticles themselves remain multi-domain at low voltages (as shown in the insets), but the magnitude of the polarization in the domains and the contrast decrease strongly with increasing λ. When λ becomes larger than 5 nm, the surface charge does not play any significant role in the screening of polarization in the 24 nm $BaTiO_3$ particles.

Thus, the FEM results confirmed the leading role of the ionic-electronic screening charges, which screen dynamically the spontaneous polarization outside the particle. When an electric field is applied to a suspension of nanoparticles, it releases some of the screening charge (mainly due to the field-induced changes of the nanoparticle polarization), leading to a decrease in the current and voltage decay rate of the cell with liquid crystal and nanoparticles (compared to the liquid crystal cell without nanoparticles).

Note that the conclusion is valid at relatively small voltages applied to the cell with a small concentration of $BaTiO_3$ nanoparticles, which agrees with the time dependences shown by the black, red, and blue curves in Figs 3a, 5a, and 5b. With increasing voltage, the nonlinear process may dominate, leading to the nonmonotonic dependence of the time decay on the concentration of nanoparticles (*e.g.*, see the sequence of black, red, and blue curves in Fig. 5c).

At the same time, the higher the concentration of nanoparticles in the liquid crystal, the greater the decrease

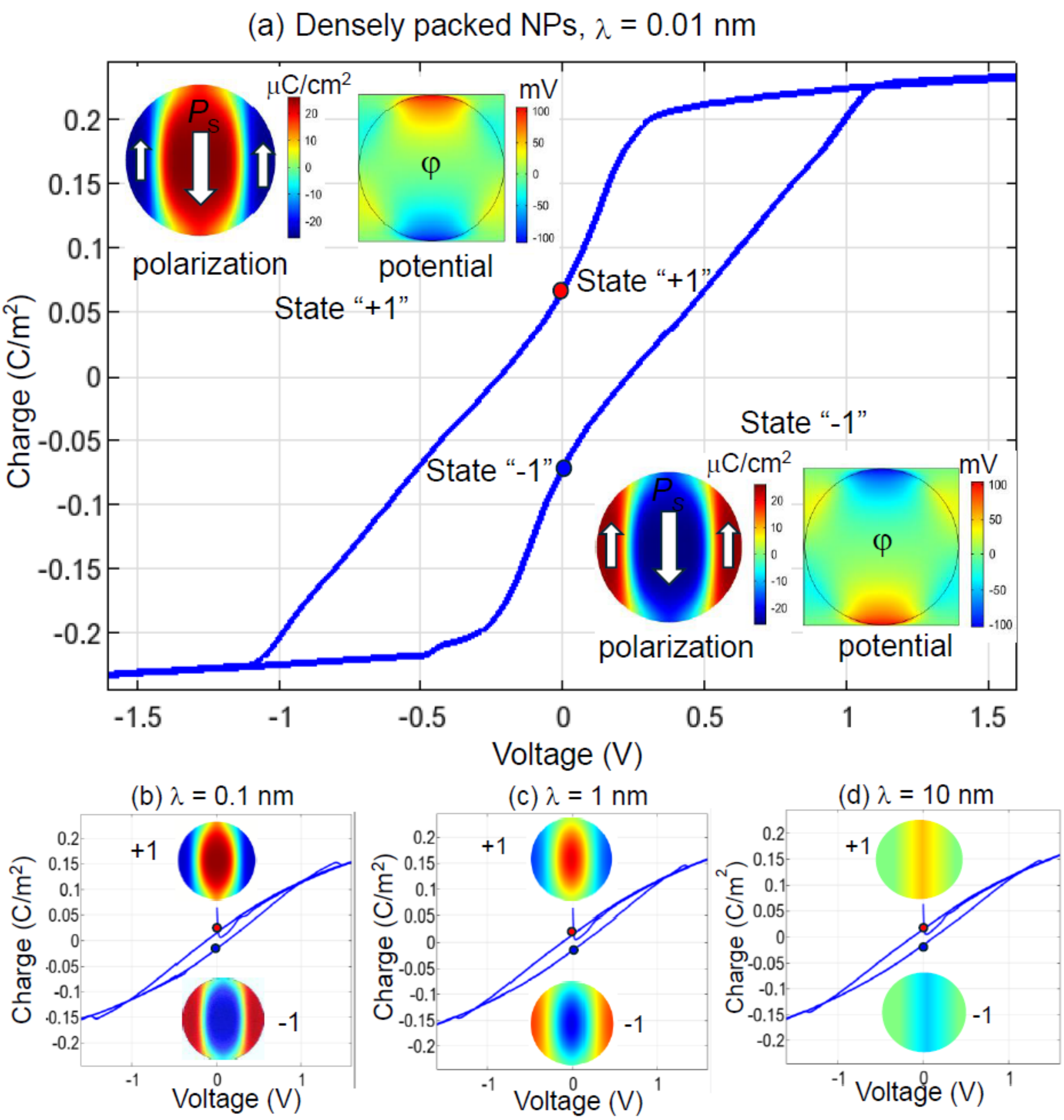

Fig. 7. (a) Dependence of the charge density on the electrodes in a cell with a liquid crystal 5CB, where spherical $BaTiO_3$ nanoparticles are densely packed. The bottom line is the distributions of the spontaneous electric polarization P_S and the electric potential φ of the $BaTiO_3$ nanoparticle in the states "+1" and "–1", which are indicated on the charge loop. Parts (b)–(d) show the dependences of the charge density on the electrodes in the cell with suspension containing 10 vol.% (b), 1 vol.% (c) and 0.1 vol.% (d) of $BaTiO_3$ nanoparticles, respectively.

in the time decay may be expected. However, one cannot expect a linear dependence of the time decay on the concentration of nanoparticles in the liquid crystal, since the process of surface adsorption of the ionic-electronic charge and its interaction with the polarization of the particles is strongly nonlinear. The indirect dipole-dipole interaction of nanoparticles in the liquid crystal is also nonlinear. With increasing the concentration of particles (more than 10%), saturation of the dependence of the time decay on the concentration of nanoparticles is possible.

4. Conclusions

The work analyses the electrophysical properties of the cells filled with pure nematic liquid crystal 5CB and a liquid crystal suspension containing 0.5 and 1 wt.% of $BaTiO_3$ nanoparticles.

Specifically, we analyze the time dependences of the current flowing through the sample at fixed values of the applied electric voltage and voltage decay across the sample in the no-load mode after switching off.

The time dependences of the current and voltage show a retarding decay rate. For samples with $BaTiO_3$ nanoparticles, a retarding decay rate is characteristic. Possible physical reasons for this are the influence of screening charges, which cover ferroelectric nanoparticles and are polarized in the external electric field, and slow ionic transport in the liquid crystal.

FEM of the polarization distribution, domain structure dynamics, and charge state of nanoparticles, performed using the LGD approach, confirmed the leading role of the screening charges, because the surface of a polarized ferroelectric nanoparticle adsorbs an ionic-electronic charge that partially screens its electric polarization in single-domain and/or poly-domain states. When an electric field is applied to the nanoparticles, it releases some of the screening charge due to the change in their polarization, leading to elongation of the time decay of current and voltage on a cell with liquid crystal and nanoparticles.

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Authors and CV

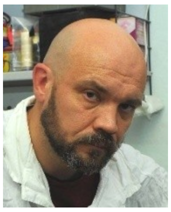
Juliya Mykolayivna Gudenko, PhD in Physics and Mathematics (Solid State Physics, 2015), Researcher at the Department of Solid State Electronics, Institute of Physics. Author of more than 10 scientific publications. The area of her scientific interests includes transport properties of low-dimensional semiconductor structures and nanoscale materials. E-mail: Gudenko.juliya@gmail.com, https://orcid.org/0009-0005-5938-5480

Oleksandr Sergiyovych Pylypchuk, PhD in Physics and Mathematics (Solid State Physics, 2015), Senior Researcher at the Department of Solid State Electronics, Institute of Physics. Author of more than 40 scientific publications. The area of his scientific interests includes transport properties of low-dimensional semiconductor structures and nanoscale materials. E-mail: pylypchuk@iop.kiev.ua, https://orcid.org/0000-0003-0136-0799

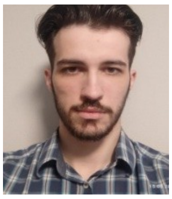
Viktor Volodymyrovich Vainberg, Doctor of Sciences in Physics and Mathematics (Solid State Physics, 2015), Leading Researcher at the Department of Solid State Electronics, Institute of Physics. Author of more than 100 scientific publications. His current research trend is transport properties of low-dimensional semiconductor structures and nanoscale materials. https://orcid.org/0000-0002-9840-8033

Vladimir Nikolayevich Poroshin, Doctor of Sciences in Physics and Mathematics (2006), Deputy Director of Research, Institute of Physics, Head of the Department of Solid State Electronics. Authored more than 100 publications. The area of his scientific interests includes fundamental research of electron and phonon transport phenomena, magneto-transport of carriers in bulk semiconductors and quantum heterostructures, effects of light absorption and emission by hot carriers. E-mail: poroshin@iop.kiev.ua, https://orcid.org/0000-0001-8217-3949

Denis Olegovych Stetsenko, PhD student, Engineer at the Department of Physics of Magnetic Phenomena, Institute of Physics. The area of his scientific interests includes magnetic phenomena in solids. E-mail: wh.denis.stetsenko@gmail.com, https://orcid.org/0009-0002-9141-0939



Igor Anatoliyovych Gvozdovskyy, PhD in Physics and Mathematics (Molecular and Liquid Crystal Physics, 2004), Senior Researcher at the Department of Optical Quantum Electronics, Institute of Physics. Author of 51 scientific publications. The area of his interests is blue phases of pure cholesteric liquid crystals doped by photosensitive chiral dopants, reactive mesogen monomers and various nanoparticle types. In recent years, he studies chiral nematic twist-bend and chiral nematic ferroelectric phases.E-mail: igvozd@gmail.com, https://orcid.org/0000-0002-4531-9942



Oleksii Vitaliyovych Bereznykov, PhD student. He is Engineer at the Department of Physics of Magnetic Phenomena of the Institute of Physics. E-mail: alexber36@gmail.com, https://orcid.org/0000-0001-6394-7304

Serhii Eduardovych Ivanchenko, PhD in Engineering Science (2023). Researcher at the Department of Physical Chemistry and Technology of Nanostructured Ceramics and Nanocomposites, Frantsevich Institute for Problems of Materials Science. Author of more than 50 scientific publications. The areas of his scientific interest include rheology, powder metallurgy, tape casting, polymer-ceramic composites, and multilayer composites. E-mail: sergonische@gmail.com, s.ivanchenko@ipms.kyiv.ua, https://orcid.org/0000-0002-9859-1835

Eugene Anatolievich Eliseev, PhD in Solid State Physics (2003), Doctor of Sciences in Solid State Physics (2009), Senior Researcher (2015), Head of the Department of Functional Oxide Materials, Department of Solid State Electronics, Institute of Physics. Author of more than 350 scientific publications. The area of his scientific interests is theory of ferroics and phase diagrams, mixed ionic-electronic conductors and related materials.
https://orcid.org/0000-0001-8124-8857

Anna Nikolaevna Morozovska, PhD in Optics and Laser Physics (2004), Doctor of Sciences in Solid State Physics (2009), Professor in Physics and Astronomy (2023), Leading Scientific Researcher at the Department of Physics of Magnetic Phenomena at the Institute of Physics. Author of more than 400 scientific publications. The area of her scientific interests is theory of ferroics and multiferroics, mixed ionic-electronic conductors and related materials.
https://orcid.org/0000-0002-8505-458X

Authors' contributions

Gudenko Ju.M.: visualization, investigation, formal analysis, writing – review & editing.
Pylypchuk O.S.: visualization, investigation, formal analysis, methodology, writing – original draft, writing – review & editing.
Vainberg V.V.: validation, methodology, conceptualization, writing – review & editing.
Stetsenko D.O.: investigation, resources.
Poroshin V.N.: validation, conceptualization, data curation.
Gvozdovskyy I.A.: investigation, resources.
Bereznykov O.V.: investigation.
Ivanchenko S.E.: resources.
Eliseev E.A.: investigation, visualization, validation, formal analysis.
Morozovska A.N.: investigation, visualization, formal analysis, conceptualization, methodology, writing – original draft, review & editing.

Динаміка зарядових станів на поверхні сегнетоелектричної наночастинки в рідкому кристалі

Ю.М. Гуденко, О.С. Пилипчук, В.В. Вайнберг, В.М. Порошин, Д.О. Стеценко, І.А. Гвоздовський,
О.В. Березников, С.Е. Іванченко, Є.А. Єлісєєв, Г.М. Морозовська

Анотація. Рідкий кристал із диспергованими сегнетоелектричними наночастинками є цікавим об'єктом для фундаментальних досліджень диполь-дипольних взаємодій далекої дії. Також він перспективний для оптичних, оптоелектронних та електрохімічних застосувань. Такі суспензії можуть служити базовими елементами для сучасних енергонезалежних комірок пам'яті та пристроїв накопичення енергії. У роботі досліджуються комірки, заповнені нематичним рідким кристалом 5CB, та комірки, що містять 5CB з наночастинками $BaTiO_3$ концентрацією 0,5 та 1 мас.% із середнім розміром 24 нм. Ми проаналізували часові залежності струму, що протікає крізь комірки, при постійній прикладеній напрузі, та динаміку напруги в режимі без навантаження. Часові залежності струму та напруги показують уповільнення швидкості спадання. Для комірок з наночастинками $BaTiO_3$ характерним є зменшення часу спадання. Можливою фізичною причиною уповільнення швидкості часу спадання є непрямий вплив екрануючих зарядів, що покривають сегнетоелектричні наночастинки, та повільний іонний транспорт в рідкому кристалі. Для пояснення динаміки струму та напруги було проведено моделювання розподілу поляризації, динаміки доменної структури та зарядового стану наночастинок у рідкому кристалі методом скінченних різниць з використанням феноменологічного підходу Ландау–Гінзбурга–Девоншира. Теоретичні результати підтвердили провідну роль екрануючих зарядів, оскільки поверхня сегнетоелектричної наночастинки адсорбує іонно-електронний заряд, який частково екранує її спонтанну поляризацію в однодоменному та/або полідоменному станах. Коли до рідкого кристала з наночастинками прикладається електричне поле, він може вивільнити частину екрануючого заряду (головним чином через зміну поляризації наночастинки), що приведе до зменшення часу спаду залежностей струму та напруги.

Ключові слова: сегнетоелектричні наночастинки, зарядові стани, іонно-електронне екранування, фероіонний зв'язок, рідкий кристал.